

(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau



(43) International Publication Date
3 May 2001 (03.05.2001)

PCT

(10) International Publication Number
WO 01/30927 A2

- (51) International Patent Classification⁷: C09D 167/06
- (21) International Application Number: PCT/IB00/01510
- (22) International Filing Date: 20 October 2000 (20.10.2000)
- (25) Filing Language: English
- (26) Publication Language: English
- (30) Priority Data:
99/6796 28 October 1999 (28.10.1999) ZA
- (71) Applicant (for all designated States except US, ZA):
BUILDTECH LIMITED [—/—]; Akara Building, 24 De Castro Street, Wickhams Cay I, Road Town (VG).
- (71) Applicant (for ZA only): **BALMORAL TECHNOLOGIES (PROPRIETARY) LIMITED** [ZA/ZA]; Building 16, CSIR Campus, Meiring Naude Road, Scientia, 0002 Pretoria (ZA).
- (72) Inventor; and
- (75) Inventor/Applicant (for US only): **SYMONS, Michael, Windsor** [GB/ZA]; 16 Luipaards Avenue, Monument Park, 0002 Pretoria (ZA).
- (84) Designated States (national): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.
- (84) Designated States (regional): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW). Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM). European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE). OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).
- Published:**
— Without international search report and to be republished upon receipt of that report.
- For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.



WO 01/30927 A2

(54) Title: METHOD OF APPLYING A POWDER COATING TO A NON-METALLIC SUBSTRATE

(57) Abstract: A method of applying a powder coating to a non-metallic substrate such as a length of a lignocellulosic material, e.g. a length of wood, is disclosed. The substrate is impregnated or has applied to the surface thereof, a composition containing a pre-accelerated, unsaturated polyester resin containing a latent catalyst therefor, either dissolved in a suitable non-aqueous solvent or mixed or dispersed in water. Thereafter the non-aqueous solvent or water is removed, and the substrate is placed in an electrostatic field or in a fluidised bed and a powder coating composition is applied thereto. The substrate is then subjected to an elevated temperature to polymerise and/or cross-link the polyester resin in or on the substrate and to cure the powder coating composition to form the powder coating. The result is a product with a decorative powder coating which may be used for many purposes.

METHOD OF APPLYING A POWDER COATING TO A
NON-METALLIC SUBSTRATE

BACKGROUND OF THE INVENTION

This invention relates to a method of applying a powder coating to a non-metallic substrate, such as for example cement bound particle board.

Powder coating is the term given to the application of a decorative coating principally to metallic articles. The coating is applied to the article in an electrostatic field by propelling dry pigmented particles from a special gun, which is friction or electrostatically activated, towards the article, the particles being attracted to the article by electrostatic forces. The particles adhere to the surface of the article, and depending upon the force of the electrostatic field, successive particles adhere until the required build up is achieved, whereafter any surplus powder falls from the article and may be recovered. The article is then moved through a suitable oven at

CONFIRMATION COPY

elevated temperatures, usually in the range of 140°C, to 185°C, or at lower temperatures in the presence of ultra violet light, to cause the powder particles to melt, flow, coalesce and cure to form a coating.

The advantages of powder coating are that a wide variety of textures and surface finishes may be achieved, the coatings are very tough and resistant to wear and in exterior grades, resistant to weathering. In addition, the powder coating method is solventless and because the powder can be recovered for reuse, wastage is virtually nil. The thickness of the coating on the article may be very accurately controlled. Further, the method is of particular application to articles of complex shape. Powder coatings are further characterised by their flexibility and adhesion so that, after powder coating, an article such as a flat sheet may be post formed over curves or edges.

One powder coating technique requires that the article to be coated must be able to sustain an electrostatic field for the particles of the powder coating composition to adhere thereto. It is possible that an article which does not retain an electrostatic field could be dampened or wetted in order for the particles of the powder coating composition to adhere to the article. However, oven heating of the article may lead to the commencement of decomposition, or to "blowing" as gasses escape through the coalescing powder film from the heated article. The alternative is fusion coating wherein the article is preheated before applying the powder coating, such as in a fluidized bed.

PCT Patent Application No PCT/GB 97/01464 (WO 97/45591) to Windsor Technologies Limited teaches a method of applying a powder coating to a length of a lignocellulosic material. The method includes the steps of impregnating the material with an impregnating composition comprising either a dicarboxylic anhydride or a tricarboxylic anhydride dissolved in a suitable non-aqueous solvent, or an isocyanate thermosetting resin dissolved in a suitable non-aqueous solvent, or a combination of the two; if necessary removing from the impregnated length of material any excess of the impregnating composition; removing the non-aqueous solvent or solvents; placing the impregnated length of lignocellulosic material in an electrostatic field or in a fluidised bed and applying a powder coating composition thereto so that the powder coating composition adheres thereto; and then subjecting the length of

lignocellulosic material to elevated temperatures to polymerise and/or cross-link the resin or resins in the length of lignocellulosic material and to cure the powder coating composition to form the powder coating.

This method can suffer the disadvantage of carbon dioxide emission as a by-product of the chemical reaction between an isocyanate thermosetting resin and either water or the celluloses.

UK Patent No 1,348,272 teaches the powder coating of a length of paper while still damp with abrasion resistant particles, such as quartz or corundum, in which the electrostatic attraction is achieved by the presence of water. This however necessitates the drying of the material before the curing of the either pre- or post-applied thermosetting resin.

European Patent Application No 636669 to DSM N.V. teaches a radiation curable binder composition for powder paint formulations. The binder composition comprises a polymer having a degree of unsaturation between 300 and 1800 grams per mole of unsaturated group (WPU), having a molecular weight M_n between 800 and 6800 and a viscosity between 1 dPas and 800 dPas, and a cross-linking agent having vinyl ether, vinyl ester or (meth)acrylate functional groups. The polymer is preferably an unsaturated polyester and/or an unsaturated polyacrylate. The binder composition can be used in the preparation of powder paint formulations which can be applied to heat-sensitive substrates such as wood and plastic and which can be cured with UV or electron beam radiation.

There is a need for a method by which a non-metallic substrate can be powder coated.

SUMMARY OF THE INVENTION

According to the invention there is provided a method of applying a powder coating to a non-metallic substrate which includes the steps of:

- (a) impregnating the substrate or applying to the surface of the substrate a composition containing a polyester resin which is selected from:

- (i) a pre-accelerated, unsaturated polyester resin containing a latent catalyst therefor, dissolved in a suitable non-aqueous solvent;
and
- (ii) a pre-accelerated , unsaturated polyester resin containing a latent catalyst therefor, mixed or dispersed in water;
- (b) removing the non-aqueous solvent or water;
- (c) placing the substrate from step (b) in an electrostatic field or in a fluidised bed and applying a powder coating composition to the substrate so that the powder coating composition adheres to the substrate; and
- (d) then subjecting the substrate of step (c) to an elevated temperature to polymerise and/or cross-link the polyester resin in or on the substrate and to cure the powder coating composition to form the powder coating.

The non-metallic substrate may be for example a length of a lignocellulosic material, e.g a length of paper or cardboard or wood; a length of a fibre board such as medium density fibre board, high density fibre board, or a fibre reinforced phenolic composite; a cementitious product such as a fibre cement or cement bound particle board, or a cement expanded mineral composite product, or the like.

The method of the invention may include various optional steps:

- (e) after step (b) and before step (c) pre-heating the substrate; or
- (f) after step (b) and before step (c) subjecting the substrate to a vacuum; or
- (g) after step (b) and before step (c) pre-heating the substrate and then subjecting the substrate to a vacuum.

In step (a) the composition may include a dicarboxylic anhydride or a tricarboxylic anhydride dissolved in a suitable solvent such as a non-aqueous solvent, which may be the same as or compatible with the non-aqueous solvent for the polyester resin, or such as water.

In step (a) the composition may also include a styrene polymer dissolved in a suitable non-aqueous solvent, which may be the same as or compatible with a non-aqueous solvent for the polyester resin.

DESCRIPTION OF EMBODIMENTS

The crux of the invention is that a non-metallic substrate is either impregnated or surface coated with a polyester resin and in this way is modified so that it induces an electrostatic attraction; that the surface of the substrate is stabilised, hardened and strengthened; that the polyester resin in the treated substrate forms a chemical linkage to the applied powder coating; and that the chemical reactions involved do not emit a gas; which all contribute to the successful powder coating of the substrate.

Step (a) of the method of the invention is to impregnate the substrate or to apply to the surface of the substrate a composition comprising a pre-accelerated polyester resin containing a latent catalyst therefor, either dissolved in a suitable non-aqueous solvent or mixed or dispersed in water.

The polyester resin may be any suitable unsaturated polyester resin. An example of a suitable resin is an isophthalic neopentyl glycol unsaturated polyester resin, developed for applications where good chemical and water resistance are essential. A specific example is NCS Ultraset 993 by NCS Resins of South Africa, which is especially designed for filament winding, marine, swimming pools, and PVC laminate applications. This resin has the following properties:

Good stain and thermal shock resistance;

Good heat and chemical resistance to a wide range of aggressive environments, including alkaline conditions.

TYPICAL LIQUID PROPERTIES

PROPERTY	SPECIFICATION	NCS TEST METHOD
Viscosity @ 25°C. mPa.s	540-800	5,3
Acid value, mg KOH/g	10-16	13
Geltime @ 25°C, 2 phr* CATALYST M, and 4 phr* ACCELERATOR ULTRACURE AC9 minutes	7-11	8
Liquid appearance	Clear pale yellow	2
Stability in the dark @ 25°C. months	5 minimum	4.1

* = parts per hundred resin, by mass.

Another option is to combine a rigid polyester resin such as NCS 33410 which is a rigid isophthalic resin with an adipic acid modified unsaturated polyester resin, particularly the isophthalics such as 8130 by NCS Resins of South Africa, added in an amount of up 50% by weight of the rigid polyester resin, so as to render the resin more flexible. The properties of cured 8130 are:

Typical Properties of Cured POLYLITE 8130 (unfilled casting)	
Prepared by catalysing with benzoyl peroxide and step cured to final post cure of 2 hours at 121°C. Tested in accordance with SATM D638-80, D790-80, D2240, ISO R75	
Shore D hardness	74
Deflection of temperature under load (1,80 mPa), °C	Yields
Elongation of break*, %	50
Tensile strength, mPa	14
Flexural strength, mPa	Yields
Flexural modulus, mPa	Yields

*Filtered resin, void-free casting

The unsaturated polyester resin must be pre-accelerated. The resin is pre-accelerated typically with a cobalt naphthanate or a cobalt octoate in white spirits, in an amount of from 0,25% to 1,5% inclusive by weight on the weight of the resin. The resin is thereafter catalysed with a latent catalyst which will decompose and trigger polymerisation at temperatures in the region of 60°C upwards, more typically above 80°C. Examples of suitable latent catalysts are a di-tertiary-butyl peroxy 3,3,5-trimethyl cyclohexane in tri-butyl phthalate such as Triganox 29B 240 or Triganox 29B 50 by Akzo Chemie, and benzoyl peroxide formulations such as tert-butyl perbenzoate, sold by Interlox under the codes TBTB and others.

The unsaturated polyester resin may be dissolved in a suitable non-aqueous solvent. The choice of solvent is dictated by cost, toxicity, evaporative rate and compatibility with the other components of the composition. Examples of suitable non-aqueous solvents are dichloromethane and acetone.

In the alternative, the unsaturated polyester resin may be one which is dispersible or miscible in water, in which case water may be used as the carrier.

The composition preferably contains the polyester resin in an amount of from 15% to 40% inclusive of the polyester resin by weight of the composition. The remainder of the composition is made up of the non-aqueous solvent or water; optionally an

additional component such as an anhydride or a styrene polymer as is discussed in more detail below; the pre-accelerator; and the catalyst. The catalyst is preferably present in an amount of from 0,2% to 0,8% by weight of the composition.

The composition may also include a dicarboxylic anhydride selected from the group consisting of maleic anhydride, phthalic anhydride, succinic anhydride and tetrahydrophthalic anhydride, or a tricarboxylic anhydride such as trimellitic anhydride. Preferably the anhydride is maleic anhydride.

The anhydride is dissolved in a suitable solvent which must be compatible with the solvent for the resin, or water. Preferably, the anhydride is dissolved in the same solvent as the resin, for example dichloromethane or acetone.

The purpose of the anhydride is to maximise the electrostatic attraction of the treated non-metallic substrate. It is also chosen for compatibility with the polyester resin.

The preferred anhydride is maleic anhydride which has a di-electric loss factor of 0,97 in dichloromethane.

The anhydride is preferably present in an amount of from 1% to 6%, more preferably from 1 to 4%, by weight of the composition.

The composition may also include a styrene polymer, e.g a styrene homopolymer, copolymer or terpolymer.

Thus, the styrene polymer may be a homopolymer, e.g polystyrene, or a copolymer such as styrene acrylonitrile, or styrene polybutadiene, or a terpolymer such as acrylonitrile polybutadiene styrene, or a blend between them.

The use of a styrene polymer imposes resistance to water uptake and to water ingress on the treated material, minimises dimensional change, particularly shrinkage on drying, improves hardness. In addition, styrene polymers have high resistance to chemicals.

The homopolymer polystyrene itself is very hard. Polystyrene has a specific gravity of 1,05 to 1,07 and a percentage water absorption after 24 hours of immersion of

between 0,04 and 0,06%. The copolymerisation of styrene with another monomer imposes special properties such as toughness. Again, the styrene polymer may be dissolved in any suitable non-aqueous solvent which may be the same as, or compatible with, the solvent used for the polyester resin. Thus, the styrene polymer may be dissolved in dichloromethane or acetone, or may be dissolved in toluene to render it compatible with the polyester resin composition.

The styrene polymer is preferably present in an amount of from 1% to 5% by weight of the composition.

The substrate may be impregnated with the composition in any suitable manner. Examples of methods of impregnation are to be found in PCT/GB 97/00440 (WO 97/32074) and PCT/GB 97/01464 (WO 97/44591), both of which are incorporated herein by reference.

For example, the impregnation may be carried out by irrigating the substrate with the composition or by immersing the substrate in the composition.

Alternatively, the substrate may simply be surface coated with the composition, again in any suitable manner, such as for example by spraying.

After step (a), if necessary there may be removed from the impregnated or coated substrate any excess of the composition.

In step (b), the non-aqueous solvent or the water is removed. This may be achieved using electronically induced heat such as infrared induced heat, or any other suitable method. When the solvent is a non-aqueous solvent, it may be recaptured for reuse.

After step (b) and before step (c), the treated substrate may be pre-heated or may be subjected to a vacuum or may be pre-heated and then subjected to a vacuum, the purpose being to de-gas and de-humidify the substrate to minimise gassing during the powder coating step.

In step (c) of the method of the invention, the substrate is placed in an electrostatic field or in a fluidised bed and a powder coating composition is applied thereto.

Generally, the powder coating composition, in the form of a finely divided pre-formulated dry powder, is propelled towards the surface of the substrate from a suitably charged applicator gun, either friction or electrostatic, such that the particles of the powder coating composition adhere to the surface of the substrate. Electrostatic charged guns are preferred such as the Super Carona by Gema. Any particles of the powder coating composition that do not adhere to the surface of the substrate, fall from the substrate and may be recovered.

Examples of suitable powders are polyurethanes or epoxy polyesters for interior use or pure polyesters for exterior use, in gloss, suede or matte, in textures, hammer tones, metallics, pearlescents, wrinkle finishes or multi colours. Curing temperatures are from as low as 100°C in the presence of ultra violet light using photosensitive catalysis, or in the range of 140°C -185°C. with a cure time of 5 to 15 minutes.

In step (d) of the method of the invention, the substrate is subjected to an elevated temperature to polymerise and/or cross-link the polyester resin in or on the substrate and to cure the powder coating composition to form the powder coating.

For example, the length of the substrate may be passed through a space heater in which the temperature of the substrate is raised to a level above 140°C, more usually up to 185°C.

A preferred form of heating is surface heating using infrared.

At the conclusion of the heating step, the powder coating composition is fully cured.

An example of the invention will now be given.

A typical formulation of a composition for impregnation or surface coating of a substrate is as follows:

Acetone	350
NCS Ultraset 993 accelerated with cobalt naphthanate	80
Triganox 29B50	2
Maleic anhydride	<u>12</u>
Total	<u>444</u>

(All parts by weight)

A substrate such as a length of medium density fibre board is impregnated with the above composition and any excess of the composition is then removed.

Thereafter, the acetone is removed by heating.

Thereafter, the treated substrate is powder coated with a suitable powder coating composition and the polyester resin and powder coating are then set and cured.

The result is a length of medium density fibre board with a decorative powder coating, which may be used for the formation of wall units or cupboards or the like.

Other products that may be produced by the method of the invention include roof sheeting, architectural mouldings, materials for use for lamination to other substrates, and the like.

CLAIMS

- 1 A method of applying a powder coating to a non-metallic substrate including the steps of:
 - (a) impregnating the substrate or applying to the surface of the substrate a composition containing a polyester resin which is selected from:
 - (i) a pre-accelerated, unsaturated polyester resin containing a latent catalyst therefor, dissolved in a suitable non-aqueous solvent;
and
 - (ii) a pre-accelerated, unsaturated polyester resin containing a latent catalyst therefor, mixed or dispersed in water;
 - (b) removing the non-aqueous solvent or water;
 - (c) placing the substrate from step (b) in an electrostatic field or in a fluidised bed and applying a powder coating composition to the substrate so that the powder coating composition adheres to the substrate; and
 - (d) then subjecting the substrate of step (c) to an elevated temperature to polymerise and/or cross-link the polyester resin in or on the substrate and to cure the powder coating composition to form the powder coating.
- 2 A method according to claim 1 wherein the non-metallic substrate is selected from the group consisting of a length of a lignocellulosic material, a length of a fibre board, and a cementitious product.
- 3 A method according to claim 1 or claim 2 which includes the step of:
 - (e) after step (b) and before step (c) pre-heating the substrate.
- 4 A method according to claim 1 or claim 2 which includes the step of:
 - (f) after step (b) and before step (c) subjecting the substrate to a vacuum.
- 5 A method according to claim 1 or claim 2 which includes the step of:
 - (g) after step (b) and before step (c), pre-heating the substrate and then subjecting the substrate to a vacuum

- 6 A method according to any one of claims 1 to 5 wherein in step (a) (i) the non-aqueous solvent is selected from dichloromethane and acetone.
- 7 A method according to any one of claims 1 to 6 wherein in step (a) the composition contains the polyester resin in an amount of from 15% to 40% inclusive of the resin by weight of the composition.
- 8 A method according to claim 7 wherein in step (a) the composition contains the latent catalyst in an amount of from 0,2% to 0,8% inclusive of the latent catalyst by weight of the composition.
- 9 A method according to any one of claims 1 to 8 wherein in step (a) the composition includes a dicarboxylic anhydride or a tricarboxylic anhydride dissolved in a solvent.
- 10 A method according to claim 9 wherein the solvent selected from a non-aqueous solvent which is the same as or compatible with the non-aqueous solvent for the polyester resin, and water.
- 11 A method according to claim 9 or claim 10 wherein in step (a) the composition contains the dicarboxylic anhydride or tricarboxylic anhydride in an amount of from 1% to 6% inclusive of the anhydride by weight of the composition.
- 12 A method according to claim 11 wherein in step (a) the composition contains maleic anhydride in an amount of from 1% to 4% inclusive of the anhydride by weight of the composition.
- 13 A method according to any one of claims 1 to 12 wherein in step (a) the composition includes a styrene polymer dissolved in a solvent.
- 14 A method according to claim 13 wherein in step (a) the composition contains the styrene polymer in an amount of from 1% to 5% inclusive of the styrene polymer by weight of the composition.

- 15 A method according to any one of claims 1 to 14 wherein in step (d) the substrate is subjected to an elevated temperature above 140°C for a time sufficient to polymerise and/or cross-link the polyester resin in or on the substrate and to cure the powder coating composition to form the powder coating.